Design and Operation of a Multiple Cigarette Smoking Machine

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In connection with studies of the chemical, physical, and biological properties of cigarette smoke, much thought and ingenuity have been applied during the past fifty years by many workers in the field to the development of laboratory equipment and techniques for the generation and collection of smoke. Because the composition of the products of combustion may vary with changes in the volume, duration, and other characteristics of the puff (Pfyl and Schmitt, 1927; Wenusch, 1936), different types of robot smokers have been developed in attempts to insure reproducibility through standardization of the puff. These may be divided into two categories according to whether the suction on the cigarette is maintained at a constant level during the puff (Commins, Cooper, and Lindsey, 1954; Jensen and Haley 1935; Kenyon, 1934; Mulinos and Osborne, 1934; Wenusch, 1937; Wynder, Graham, and Croninger, 1953) or whether the suction is purposely varied (Bradford, Harlan, and Hanmer, 1936; *Consumer Reports*, 1953 and 1955; Derr, Riesmeyer, and Unangst, 1937; Pfyl, 1933; Pyriki, 1934; Wolman, 1953).

It would seem that a choice between the two would depend upon which type more closely simulates human puffing. Bradford *et al* (1936), with the aid of kymographic tracings, found that the intensity of suction applied to a cigarette varies during human smoking.

Table 1. Puff Measurements on Twenty Male Subjects

	Subject	Average Puff Volume (cc) (Air Intake)	Average Puff Duration (sec.)	Average Maximum Suction During Puff (in. water)
1.	REM	22.3	0.97	8.2
2.	LQH	47.3	1.35	13.3
3.	MCT	41.9	2.22	7.7
4.	PSK	33.3	1.60	9.9
5.	JLE	35.6	1.82	6.3
6.	DTW	48.4	1.43	13.0
7.	WJC	56.0	1.94	12.5
8.	URB	33.8	1.75	7.7
9.	TBC	53.0	1.74	14.6
10.	UGB	30.0	1.26	11.1
11.	JCR	37.2	2.32	9.4
12.	WBL	36.3	1.58	9.2
13.	CCJ	55.5	1.17	19.1
14.	REN	37.8	1.74	8.8
15.	PCM	39.6	2.03	5.8
16.	CLD	23.9	1.25	10.3
17.	GHB	23.2	1.22	7.1
18.	GSG	46.8	1.65	12.8
19.	MAS	22.9	1.23	8.9
20.	CWG	23.2	1.41	7.8
Average of All Subjects		37.4	1.58	10.2
Standard Deviation		±10.7	±0.37	±3.2

Average Puff of Person With Puff of
(a) Lowest Average Volume = 22.3 cc.
(b) Highest Average Volume = 56.0 cc.
(c) Shortest Average Duration = 0.97 sec.
(d) Longest Average Duration = 2.32 sec
(e) Lowest Average Maximum Suction = 5.8″ water
(f) Highest Average Maximum Suction = 19.1″ water

Materials and Methods

Studies made in the present investigation demonstrate that each smoker has his own, almost unique, puffing pattern, within which he may show considerable fluctuation with respect to volume, duration, and frequency of puffing. For obtaining kymographs of human puffs, a port between the mouthpiece and cigarette end of a cigarette holder was fitted into the stem of the holder. Light rubber tubing was used to connect the port with the kymograph. For obtaining volume measurements, the cigarette holder was fitted into the volume measuring device illustrated in figure 5 and described further on. To minimize subjective errors, the subjects were faced away from the instruments and were not allowed to know which of their puffs were being recorded. Typical kymograph curves are shown in figure 1; data on volume and duration of puff are given in table 1. Among the subjects tested, several were found who maintained the suction on the cigarette constant over a large portion of the puff, some who varied the suction in a rapid, erratic manner, and others who varied the suction smoothly.

A composite of the kymograph

curves obtained is very similar to the curve illustrated in figure 9. However, such a curve is not particularly descriptive of that of any individual subject tested. In view of these circumstances, it may be maintained that until far more has been published about the effect of varying the volume, duration, and other characteristics of the puff upon the chemical and biological properties of the smoke, one type of smoking machine is as useful as the other, provided it has adequate capacity, that it lends itself to standardization and duplication, that it smokes every cigarette in a consistent manner, and that the characteristics of the puff lie well within the range of those encountered in human smoking.

As a needed modification of the various smoking machines described in the literature, the mechanical puffer described below was devised. Under conditions which control the flow of air through individual cigarettes, it is capable of handling a larger number of cigarettes than any smoking machine heretofore described. As set up in this laboratory, it smokes up to twenty cigarettes at a time, and subjects each cigarette once per minute to an air intake of 35 cc. during a puff of two seconds' duration. This schedule is within the range of values obtained in a limited study of human smoking, as will be seen by reference to table 1. Except for the volume of the puff, (which is based on *air intake*), it is the schedule selected by Bradford *et al* (1936), and is that followed by most American investigators (*Consumer Reports*, 1953 and 1955; Derr *et al*, 1937; Finnegan, Fordham, Larson, and Haag, 1947; Fishel and Haskins, 1949; Haag and Larson, 1948; Rayburn, Harlan, and Hanmer, 1953; Wolman, 1953). The reason for the difference in the volume per puff will be explained below. Through relatively simple mechanical changes or adjustments, the frequency and the volume of the puff may be varied; and the duration of puff may be modified more or less, depending upon the frequency of puffing. Schematic drawings of the smoking machine in two of its forms are given in figures 2 and 7.

Each cigarette is snugly held in a circular opening in a thin rubber diaphragm stretched across the entrance to a horizontal glass helix constructed of Pyrex tubing with an internal diameter of approximately 10 mm. Details of the cigarette holder and the coil are given in figure 3. If desired, the cigarette may be inserted directly into the glass holder, in which case the rubber diaphragm is dispensed with and the cigarette is sealed into the holder with the aid of a viscous cement, free from contaminating volatile matter. A good cement for the purpose is a solution of dextrose in water sufficiently concentrated to prevent its penetration through the cigarette paper (Kenyon, 1934). Care must be taken that the inserted end of the cigarette is not unduly compressed, else, according to tests made in this laboratory, the concentration and the quantity of particulate matter in the smoke drawn from the cigarette may be reduced, and other undesirable effects, such as substantially increased resistance to air flow, incurred.

Attached to the base of the cigarette holder is the ball member of a glass ball-and-socket joint. This is used in the measurement of volume of puff, as will be described further on.

The glass coil, which comprises the smoke collecting vessel, is cooled by submersion in a bath of dry ice in acetone (temperature about —80° C.). Its outlet end is connected by

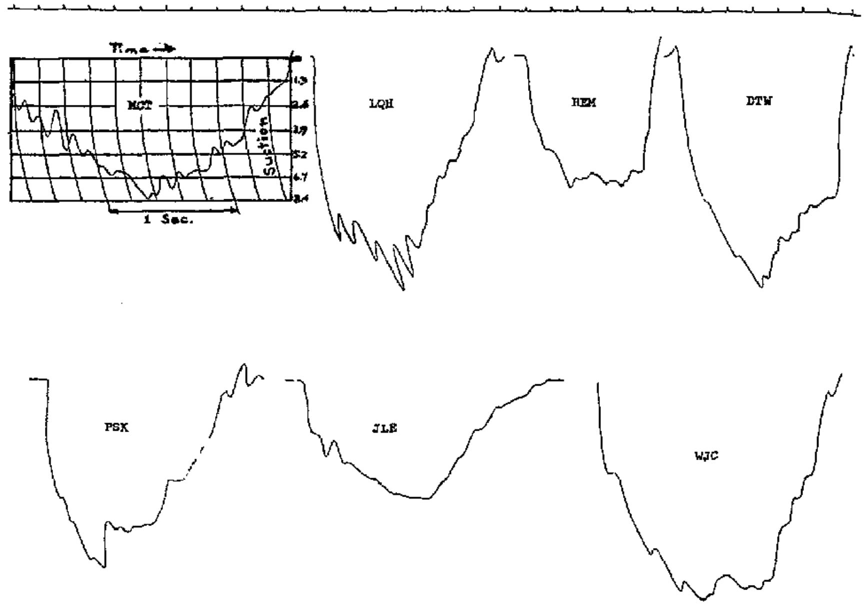


Figure 1. Kymograph Tracings of Human Puffs. (Initials in each tracing are those of the individual tested. The lines superscribed over the first tracing are suction and time coordinates. Suction is given in inches of water.)

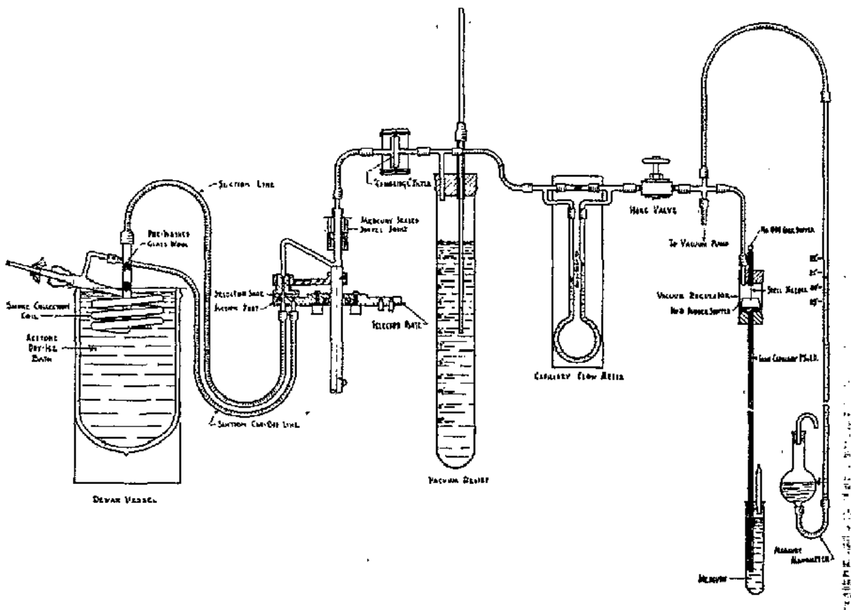


Figure 2. Schematic Drawing of Vacuum Pump Operated Form of the Smoking Machine.

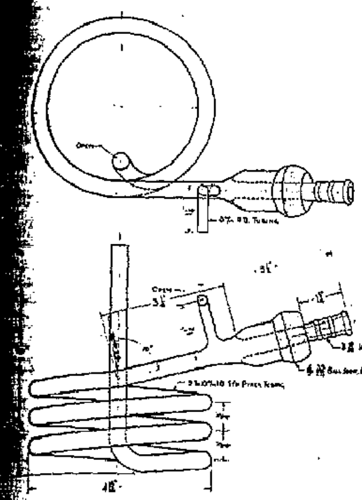


Figure 3. Cigarette Holder and Smoke Collection Coil.

means of rubber tubing to one of twenty ports in the horizontal circular selector plate of a suction timing mechanism (figure 4). Suction is applied to these ports, one after another, through a neoprene-clad rotating selector shoe which slides over the ports at a uniform rate of travel so that each cigarette in succession is subjected to suction over a two second interval, once per minute. To prevent chattering of the selector shoe, a thin film of vaseline-base stop-cock grease should be maintained on the surface of the selector plate. For perfectly flush contact between the two, the selector plate and the shoe must be finished by hand scraping, even when the selector shoe is to be clad with neoprene. In one form of smoking machine (figure 2) the suction is conveniently generated by means of a ¼ h.p. motor-driven suction pump. In another form (figure 7), the suction is applied by means of a piston oscillating in a cylinder.

Between the selector and the suction pump in the first form of the machine (figure 2) are an easily adjustable vacuum relief, a Cambridge "absolute" filter, a capillary flow meter, a Hoke valve, a suction regulator, a mercury manometer, and a two-liter trap. The function of the Cambridge filter is to protect the capillary flow meter from dust or traces of smoke in order to insure stability of calibration. The vacuum relief is necessary to prevent the suction from building up excessively between puffs, which would result in too sudden a surge of air through the cigarettes at the beginning of each puff. In the simple form illustrated in the drawing, (figure 2), the vacuum relief also serves as a water manometer, showing the suction, or drag, on any cigarette as it is being smoked. The capillary flow meter is a convenient aid in the setting of the Hoke valve to control the rate of flow of air through the cigarettes.

Operating Instructions
Suction Pump Model

At the start of a smoking run, the Hoke valve is closed, the suction regulator is adjusted to hold the vacuum at the mercury manometer at a constant level somewhere between, say 20″ to 21″ of mercury, the Hoke valve is adjusted until the reading on the capillary flow meter corresponds to a 35 cc. puff, approximately, and the adjustable vertical tube in the vacuum relief is regulated to a depth 1½ to 2½ inches greater than that necessary to prevent bubbling during the puff. The cigarettes are then lighted by means of a glowing, flat coil of electrically heated resistance wire. The Hoke valve is now readjusted, if necessary, so that the volume of the puff is 35.0 cc. ± 0.5 cc., as determined by measurements of the actual volume of air being sucked per puff into the cigarettes.

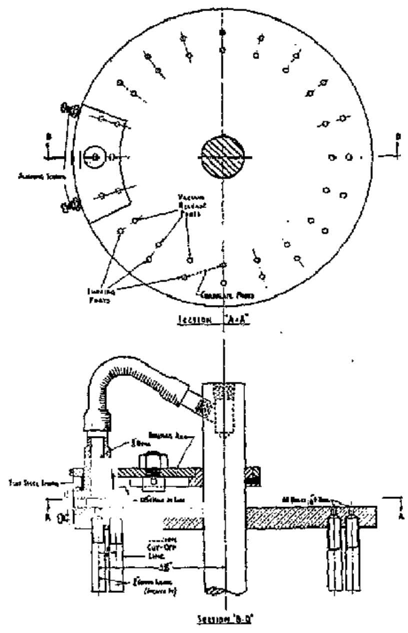


Figure 4. Suction Timing Mechanism for Vacuum Pump Operated Form of the Smoking Machine.

To measure the volume, use is made of the device shown in figure 5. This is comprised of a cylindrical vessel closed with a lightly stretched, thin rubber diaphragm on which is cemented a small mirror near the wall of the vessel. The interior of the vessel communicates by means of a rubber tube with the socket member counterpart of the ball member located at the base of the cigarette holder. Whenever a measurement of air intake is desired, the socket member is slipped over the cigarette and held snugly against the ball member just before the puff is taken. As is evident from the drawing, the air drawn through the cigarette is now taken from the cylindrical vessel, thus causing its rubber covering to be sucked inward. The extent of flexing of the diaphragm is indicated by a beam of light centered on the mirror and reflected as a spot onto a graduated scale several feet away. If the measuring device is set up according to the specifications given in the drawing, the relationship betwe-

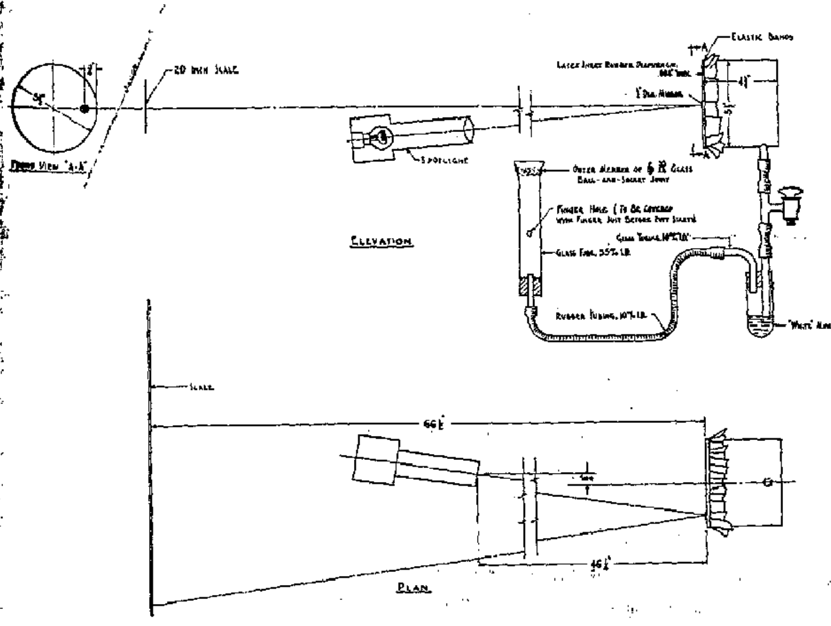


Figure 5. Volume Measuring Device.

the volume of the puff and the travel of the spot of light on the graduated scale is linear up to at least 50 cc., and the suction under the rubber diaphragm is of the order of only 3 or 4 mm. of water when 35 cc. of air have been pulled through the cigarette. About once a week, or whenever a diaphragm is replaced, the volume measuring device is calibrated with the aid of a burette. Since a check valve maintains the deflection at its maximum reading, and since the total inertia of the system is so small, this device has been found to yield reproducible results on volumes up to 50 cc. for suction intervals from 0.5 to 5 seconds, as measured by a dropping water column in an inverted burette.

It will be noted that the foregoing method of measuring the volume of a puff refers to the air entering the cigarette. On the other hand, the more common practice in the past has been to define the volume of the puff as the volume of the gaseous phase of the smoke measured after the smoke collecting trap. It would seem to matter little which definition of volume of puff is used, i.e., whether it is based on the air intake or on the volume of gas leaving the smoke collection train, so long as the details of the conditions under which the volume is measured are known. According to measurements, made with the use of a dropping water column in a calibrated burette as a puffing device, an air intake of 35 cc. into conventional cigarettes initially 70 mm. long is equivalent to a smoke gas phase volume at room temperature of approximately 42 cc.

When, for this work, a standard volume of air intake per puff of 35 cc. was chosen, it was not realized that there is so great a difference between the volume of air intake and the volume of the gas phase of cigarette smoke (at room temperature), else there would have been concern over the fact that so many investigators have used 35 cc. of the gas phase of the smoke as a standard puff. However, it is interesting to note that Pfyl (1933), who made 15 measurements on 7 persons, found an average volume of intake air of 42.5 cc.; and, the present studies, as shown in table 1, indicate a volume of 37.4 cc. as the average volume of intake air, based on multiple measurements on 20 individuals. The standard deviation of the 20 averaged values of puff volume (air intake) was ± 10.7 cc. Thus, the 35 cc. air intake chosen at the outset was well within the range of volumes for human puffing.

The cigarettes are smoked to a butt length of 23 mm., whether or not they are filter-tipped. The choice of the butt length is of some importance, since the nicotine content of the smoke increases as the butt length decreases (Jensen and Haley, 1935). A 23 mm. butt, however, appears from the literature to be fairly typical of American practice, although it is considerably longer than that usually reported by European investigators, as summarized in table 2.

The helical collection coil shown in figure 3 was designed with several objectives in view.

1. The internal volume of the coil is low, yet the completeness of condensation of the particulates within the coil is well over 95% at —80° C., as compared with total smoke collectable by filtration and condensation at — 80° C. in glass-wool packed "U" tube collection vessels. The advantage of the low volume is that it minimizes variations in the time-pressure relationship at the base of the cigarette with variations in the resistance of the cigarette to flow of

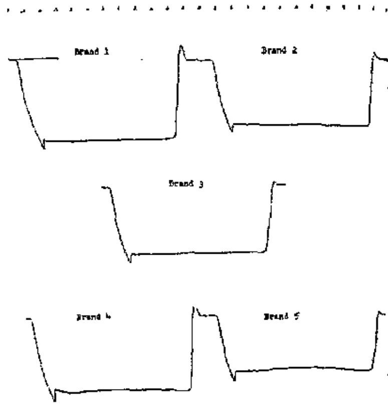


Figure 6. Kymograph Tracings of Constant Suction Puffs on Five Different Brands of Cigarettes.

Table 2. Literature on Butt Length

Reference	Butt Length
Nature of Cigarette Smoke. Bradford, Harlow, Harlan, and Hanmer (American Tobacco Company). *Ind. Eng. Chem.*, 29: 48 (1937)	23 mm.
Some Basic Facts of Medical Interest Concerning Cigarettes and Their Smoke. Haag and Larson (Medical College of Virginia). *The Interne*, page 111, April, 1948	23 mm.
Composition of Cigarette Smoke. Fishel and Haskins (Ohio State University). *Ind. Eng. Chem.*, 41: 1375 (1949)	19 mm.
Cigarettes. *Consumers Report*, 18: 68 (1953)	23 mm.
Experimental Production of Carcinoma with Cigarette Tar. Wynder, Graham, Croninger (Sloan-Kettering Institute for Cancer). *Cancer Research*, 13: 858 (1953)	20–25 mm.
A Study of Cigarettes, Cigarette Smoke and Filters Wolman (American Medical Assn.) *J. A. M. A.*, 157: 1309 (1955)	23 mm.
Tobacco Smoke. Waser and Staehli (County Laboratory, Zurich). *Z. Untersuch. Lebensm.*, 64: 477 (1932)	15 mm.
Determination of Nicotine in Tobacco Smoke. Pfyl (German Health Dept.) *Z. Untersuch. Lebensm.*, 58: 505 (1933)	15 mm.
On the Nicotine Content of the Main Stream Smoke. Wenusch (Austrian Tobacco Monopoly). *Z. Untersuch Lebensm.*, 72: 217 (1936)	13 mm.
Investigation of Tobacco Smoke (German). Pyriki. (Inst. for Tobacco Research, Wahlsdorf, Germany), *Z. Untersuch. Lebensm.*, 88: 262 (1948)	15 mm.
Determination of Nicotine and Tar in Tobacco Smoke. Staub and Furer (County Laboratory, Zurich). *Mitt. Gebiete Lebensm. u. Hyg.*, 44: 375 (1953)	20 mm.

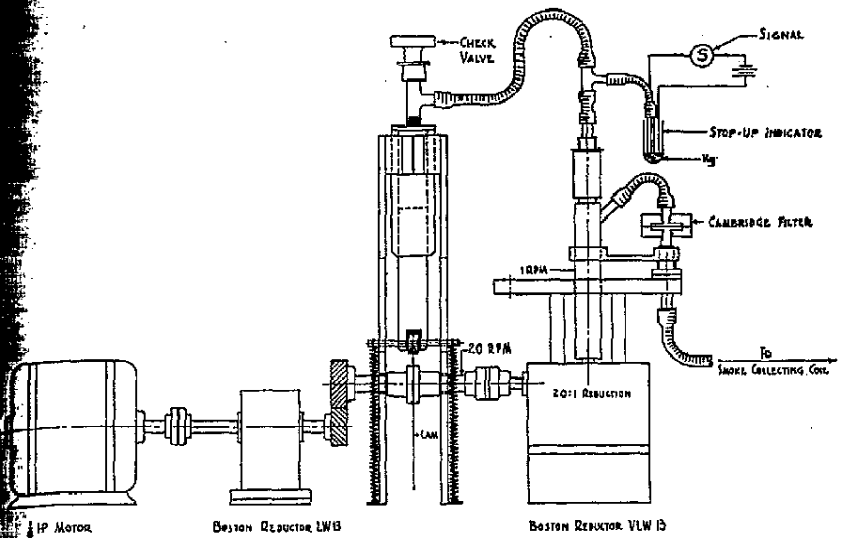


Figure 7. Schematic Drawing of Cam Driven Form of the Smoking Machine.

air or smoke.

2. The vertical riser at the outlet of the coil permits the insertion of several wisps of glass wool (preferably three, each weighing about 8 to 10 mg.) which serve to increase the completeness of collection of particulates and which later, when wet with solvent during the extraction of the smoke condensate from the coil, as described further on, catch some of the more volatile constituents of the condensate as the coil is permitted to come up to room temperature. (A good grade of glass wool for the purpose is #800 Fiber Pyrex Brand Glass #7220, Corning Class Works Catalogue No. LP 34. It must be pre-extracted to remove oils. The use of purified toluene followed with ethanol is recommended).

3. Because of its shape and low volume, the coil lends itself well to extraction of the condensate with minimum quantities of solvent.

The outlet end of each helical collection vessel, as stated above, is attached by means of flexible tubing to one of twenty ports in the circular selector plate, as shown in figure 4. Traveling across these ports is a rotating selector which presents to each port in succession a circular opening. This opening communicates through a sealed swivel to the source of suction. The diameter of the circular opening in the selector shoe, the diameter of the ports in the selector plate, and the rate of rotation of the selector on the circular selector plate manifestly determine the duration of the puff.

In order that residual suction in the collection train be relieved at the instant that the circular opening in the selector shoe completes contact with the port opening, the heel of the selector shoe at that instant uncovers a relief port in the selector plate (figure 4) which is connected by means of rubber tubing to the base of the cigarette holder, thus permitting air to rush directly into the coil, i.e., without passing through the cigarette. In this way, the stream of air flowing through the cigarette is interrupted at a definite instant; otherwise it would continue to flow through the cigarette for an appreciable, varying time interval to relieve the residual vacuum in the smoke collecting coil. Because of the small volume of the coil, the volume of air entering the smoke collecting vessel through the suction relieving port at the end of each puff varies to an unimportant extent with variations in the residual suction (less than about ¾ cc.) at the instant the suction relieving port opens.

The design of the selector shoe described above results in a substantially constant suction on the cigarette throughout the puff. This is illustrated by the kymograph curves of figure 6, obtained with a Phipps and Bird tambour, type 70-710. The slight irregularities near the start and end of the tracings are due mainly to the momentum of the kymograph pen and to the elasticity of the tambour diaphragm. It was found preferable to operate the tambour undamped. The recording drum was run at 4.1 cm. per second, as in the case of all kymograph tracings shown herein. In the tracings, the jogs in the timing line represent intervals of 2/10 second.

Because the puffing device is operated with a relatively high vacuum on the pump side of the Hoke valve, one of the most serious objections to constant suction smoking machines is minimized, namely, the tendency of the volume of the puff to fluctuate seriously with the resistance of the cigarette to the flow of air. The high vacuum makes it necessary for the operator to throttle the Hoke valve to the point where its resistance to the flow of air is very much greater than that of the cigarette. Under this condition, normal variations in the resistance to flow through any cigarette from one puff to another, or among different cigarettes, have relatively little influence on the volume of the puff. Uniformity in the volume of the puff is also favored if all cigarettes in the smoking machine at one time are taken from groups with a weight range of about 1/10 gram, e.g., 0.95 to 1.05 grams, and 1.05 to 1.15 grams for 70 mm. cigarettes without filter tips. Normally the minimum and the maximum weights of the cigarettes in a carton are within 2/10 grams of each other. Consequently the 200 cigarettes in a carton may be divided into two groups with weights in each group varying within 1/10 gram.

It is, of course, essential that the vacuum applied to the Hoke valve be maintained as closely constant as feasible. To achieve this end, the simple but effective suction regulator shown in figure 2 was improvised.

If, instead of maintaining the suction on the cigarette constant, it is desired to vary the suction during the puff according to a predetermined schedule, the neoprene facing of the selector shoe may be removed and the circular opening in the shoe may be replaced with a shaped opening of empirically determined design. In this case, the opening in the selector shoe is shaped by hand until the desired kymograph tracing is realized. This is conveniently done with the aid of Swiss pattern files.

Whether the opening in the selector shoe is circular or "shaped," measurements of the volume of the puff have shown an average deviation of about 0.4 cc. and a standard deviation of about 0.6 cc. when the machines are operated at the 35 cc. level. These deviations in volume are close to the values found for the average and standard deviations, respectively, of the *intake* volume of the puff when a dropping water column, such as described by Bradford *et al* (1936) was used to effect puffing. It is apparent from the large difference between the intake volume and the smoke gas phase volume mentioned earlier

that relatively slight variations in the amount of tobacco burned from puff to puff arising from variation in the density of the tobacco in the cigarette, or for any other reason, would tend to cause noticeable variations in the volume of air intake, even though the volume of the gas phase of the smoke is maintained constant from puff to puff.

Cam Actuated Piston Model

Another method of shaping the puff is to substitute for the suction pump, suction regulators, and Hoke valve, a cylinder provided with a piston actuated by a cam of predetermined contour. Such a layout is shown in figure 7. In this case, the opening in the neoprene-clad selector shoe is conveniently circular, and the timing is adjusted with the aid of timing screws provided on the arm which actuates the shoe, so that any port on the selector plate is in communication with the hole in the selector shoe during the interval when the piston is being displaced outwardly. A detail of the selector plate and shoe is given in figure 8. During the inward travel of the piston, air is expelled from the cylinder through the check valve shown in figure 7.

Mechanically operated piston type puffers have been brought to a high state of development by O'Keeffe and Lieser of Philip Morris, Inc., Richmond, Virginia (private communication). The machine described herein differs from theirs in that it utilizes a single piston and a cigarette selector rather than an individual puffing mechanism for each cigarette. It differs also in the mechanism used to actuate the piston in that it employs a cam whose contour can be changed over a wide range to effect changes in the characteristics of the puff, as measured kymographically, (figures 9 and 10b), even to the point of approaching a constant suction type of puff similar to those illustrated in the kymographs of figure 6.

If the kymograph tracing is to incorporate a sharp cessation of smoke flow at the end of the puff, the use of a vacuum release opening connected to the base of the cigarette holder, such as was included in the form of the smoking machine first described and shown in figure 2, may be advantageous. This is shown in the kymograph tracings of figures 10a and 10b, which were obtained respectively without and with the use of a vacuum release port.

The design of the cam is partly an empirical procedure, since it is necessary to make compromise allowances for unpredictable fluctuations in a number of important variables involved in the shaping of the puff. Chief among these are (1) the changes from puff to puff and from cigarette to cigarette in the resistance of the cigarette to the flow of smoke, and (2) variations in the increase in volume of the gas during the puff. It is also necessary to take into account the buffering effect of the smoke collection train and associated tubing as well as the magnitude of the residual vacuum in the train at the instant it is desired to terminate the puff. These are factors which one must keep clearly in mind in the design of any type of smoking machine if the effects of the unpredictable variations on the volume of the puff, the duration of the puff, and the contour of the kymograph tracing are to be distributed as desired.

Handling of Smoke Condensates

Transfer and Concentration Adjustment

On the completion of a smoking run, as many as thirty cigarettes may have been smoked in each of the twenty smoke collection coils attached to the smoking machine. If the smoke condensate is to be used for biological studies, it should be transferred from the collection vessels to proper storage containers as quickly as possible in order to minimize the duration of exposure to room temperature and to the oxidizing effects of air.

Out of an abundance of caution, before any coil is removed from the chilling bath, the lighting in the room is subdued to minimize the chance for photochemical changes in the smoke condensate.

It is convenient to make transfers from three collection coils at a time. Before the coils are removed from the chilling baths, the inlets of the coils are closed both at the cigarette holder and at the suction release side arm. As closures, ground glass stoppers may be used in the cigarette holders, and short lengths of gum rubber tubing on the suction release side arms. Two ml. of the solvents (a very satisfactory solvent is a mixture of 3 parts purified ethanol and 2 parts purified toluene by volume) are now added to the outlet end of each coil. This is more than enough to saturate the wisps of glass wool. As the coils warm up to room temperature, the gases which form or expand pass through the solvent-wet glass wool in the vertical riser at the outlet. As soon as the coils have approached room temperature, the inlet closures are taken off and any particles of tobacco which may be present in the cigarette holders are removed with the aid of swab sticks. The coils are next wiped free of the moisture which has condensed on them and are placed on a clean work bench, preferably in an area which has been covered with a clean white blotter.

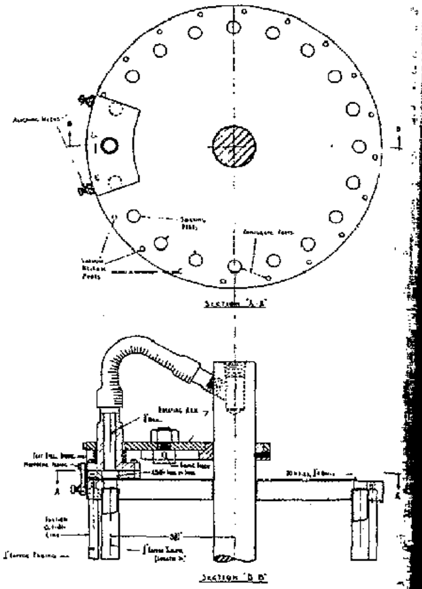
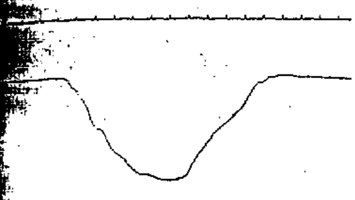


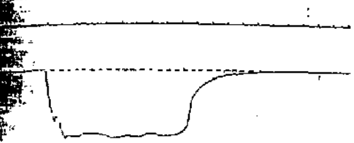
Figure 8. Suction Timing Mechanism for Cam Operated Form of the Smoking Machine.

For solution and removal of the condensate in a minimum volume of the solvent, two dropping pipettes are used. These are made of 6 mm. glass tubing, one 4" in length, the other 6" to 8" in length. Both are fitted with a 1 ml. rubber bulb. The shorter pipette is used for transferring aliquots of pure solvent to the condensate traps; the longer pipette is used for transfer of the condensate solution from the traps to a ground glass stoppered Erlenmeyer flask large enough to hold the condensate solution from all coils (figure 11).

Whatever solvent has seeped from the glass wool into the coil proper is made to run slowly through the length of the coil toward the inlet end by a tilting and turning of the coil in the proper direction. This is done with all the coils which the operator is handling at one time, and the viscous solution resulting is removed from the inlet ends of the coils with a dropping pipette and is transferred

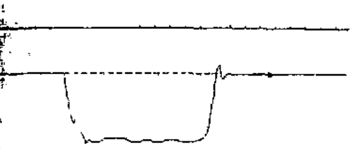


...9. Kymograph of Puff Obtained with ...le Suction Cam.



... 10a. Kymograph of Puff Obtained with ...tant Suction Cam, *Without* Use of Vac. Release.

...n for Ca... Machine.



... 10b. Kymograph of Puff Obtained with ...tant Suction Cam, *With* Use of Vacuum ...se.

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... Erlenmeyer flask. In order to ...plete the transfer with the ...llest possible volume of solvent, ...ut 0.2 ml. portions of solvent ... added through the outlet ends ... the coils, and the above proce...re followed until the glass wool ...ree of color. The wisps of glass ...ol are then removed and dis...ded.

...imilar aliquots of solvent are ... added at the inlet end of one ... the coils and passed slowly ...ough the length of the coil with ...irling motion in a manner such ...t every portion of the inner sur... of the coil is wet, until the ...id reaches the bottom of the ...tical riser at the outlet of the ... The liquid is then removed ... this coil, transferred to the ... end of the second coil, the ...ling procedure repeated, and ... solution transferred in the ... manner to the third coil. In ... way, maximum advantage is ...n of the strong dissolving ac... of the solvent. This proce...e is followed until the coils are ...fectly clean.

...If desired, the weakly colored so...tion obtained during the latter ...rt of the transfer operation may ...reserved for use in the extrac... of condensate from the next ... of coils, which in the meantime, ...e been allowed to come to room ...perature.

Throughout the entire procedure, ... Erlenmeyer flask is kept immersed in pulverized dry ice in order to minimize the chance for changes in composition of the condensate which might otherwise occur at room temperature during the transfer period. The flask should be kept closed as much of the time as possible, so that condensation of atmospheric moisture within it will be avoided.

Through the use of the technique described, a condensate equivalent in concentration to the smoke from as many as eight cigarettes per ml. of solution is attainable if the ethanol-toluene solvent mixture is used. In the adjustment of the concentration to any level below this, it is necessary to have an appropriate method of determining concentration. In order to permit adequate sampling of the solution to be tested, it is necessary that the solution be brought to room temperature for a length of time sufficient to allow it to become homogeneous in appearance when it is swirled in the Erlenmeyer flask. As soon as the sample has been taken, the Erlenmeyer flask is rechilled. This laboratory has used three methods of determining concentration, as follows:

1. *Oven Dry Residue:* Pipette one ml. of condensate solution into a small, tared weighing dish. Evaporate to near dryness on a steam bath or on the top of the drying oven. Place in a 95° C. oven and leave for 16 hours. Remove from oven and place immediately in a dessicator. Weigh after 20 to 30 minutes in the dessicator, and record the weight of the residue.

2. *Air Dry Residue:* Pipette one ml. of the solution into a tared weighing dish and evaporate to apparent dryness under a gentle stream of dry air at room temperature. Leave in a dessicator under a direct, gentle stream of dry air for 16 hours. Weigh and record weight of residue.

(As is well known to workers in this field, the weight of residues obtained on drying may vary according to the drying procedure and to the time of exposure after apparent dryness has been reached. This is because of a slow loss of components of low volatility and because of oxidation effects.)

3. *Colorimetric Method:* As standards, make up a series of solutions of smoke condensate covering the pertinent range of concentrations. The condensate used should be obtained from the blend or mixture of blends of cigarettes corresponding exactly to those to be evaluated. To a 5 ml. cuvette of a Coleman Spectrophotometer containing 5 ml. of the solvent, add by means of a calibrated micropipette 0.005 ml. of the condensate solution. Read the optical density against the solvent blank at a wave length of 375 mμ with a P. C. 4 filter. Plot the optical densities found against the air dry and the oven dry weights of the corresponding samples.

For the optical evaluation of a condensate solution of unknown concentration, use the same dilution procedure and instrumental settings, and obtain the condensate concentration from the calibration curve.

(The 375 mμ wave length used does not correspond to any maximum light absorption region and has no specificity. It was chosen simply because it represents a wave length where the slope of the absorption curve approaches zero. The optical densities obtained, although of empirical nature, will be found to yield reproducible correlations with the oven dry and air dry weights. It is necessary to use a plotted calibration curve, since Beer's law is not applicable. This is due, at least in part, to the light scattering effect caused by the fluorescence of the smoke solution.)

Once the concentration has been established, the solution is allowed to warm to room temperature and sufficient solvent is added to give the desired dilution.

Vialing and Storage

The condensate solution, adjusted to the desired concentration, should be vialed and refrigerated immediately. Flat bottom, open neck vials of desired capacity which have been washed with acetone and distilled water and have been oven-dried at 105° C. are used. To effect the transfer to the vials, the condensate solution is permitted to come to room temperature and is withdrawn from the Erlenmeyer flask into a 10 ml. graduated hypodermic syringe fitted with a 3" needle. The needle is wiped clean with absorbent paper tissue and is inserted into the body of the vial, great care being taken not to wet the inner surface of the vial neck. (The reason for this precaution is to avoid formation of any secondary combustion products when the necks are later heat-sealed.) The desired volume of the condensate solution is accurately expelled into each vial, and the needle is withdrawn with care to prevent deposition on the neck.

The vials containing the concen-

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Figure 11. Equipment for Transferring Smoke Condensate from Collection Coil.

trate are now chilled by partial immersion in pulverized carbon dioxide snow. As soon as the contents have congealed, the vials are flushed with pure nitrogen gas introduced through a 3" hypodermic needle. While still in the carbon dioxide snow, the vials are sealed with a small oxygen-gas flame. They are then stored at a temperature of —60° C. or lower until ready for use.

Typical Smoke Collection Data

In the preparation of cigarette smoke condensate, equal numbers of the five leading brands of regular size (70 mm.) non-filter tipped cigarettes were smoked individually in a regular rotation by brand. The cigarettes were bought by the carton on the open market from dealers having a moderately high turnover, and were smoked within two weeks of purchase. No special selection of cigarettes was made according to weight, moisture content, or the like, i.e., they were used as found, except that each carton was divided into two weight groups, as explained earlier, and only cigarettes of one group and of one brand were in the smoking machine at one time. The constant suction puff was used.

Under the conditions of smoke condensate collection (about —80° C.), and with cigarettes of current production, approximately 70 mg. of condensate per cigarette were deposited in the collection vessel. This represents a collection efficiency of well over 95 per cent of the smoke constituents that were condensable under ideal conditions at this temperature. Of this material, close to 92 per cent, by weight, was recovered during the transfer to the vials. The other 8 per cent represents a loss of the most volatile constituents.

The concentration of the solution was adjusted to be equivalent to five cigarettes per ml. rather than to a definite weight of smoke condensate per unit volume.

The solution was transferred in 3.0 ml. aliquots to clean 5 ml. ampoules which were labeled with the date of preparation and a reference code and then sealed and stored in the manner explained above.

Of the solution in the vials, about 42 to 48 mg. per cigarette were non-volatile when the solution was tested by evaporation to apparent dryness under a gentle stream of dry air at room temperature and the residue was maintained in the stream of dry air for 16 hours; 27 to 31 mg. were non-volatile when the solution, evaporated to apparent dryness, was oven-dried at 95° C. for 16 hours.

It must, of course, be recognized that not all components of smoke are condensed by the method used. "Permanent gases" escape as well as small portions of the more volatile components.

Summary

Described is a multiple cigarette smoking machine designed for the preparation of relatively large quantities of cigarette smoke condensate suitable for chemical and biological studies. Methods are discussed for the control and measurement of volume of puff and other puff characteristics. A procedure is given for the transfer of cigarette smoke condensate from collection vessels to vials in a highly concentrated solution.

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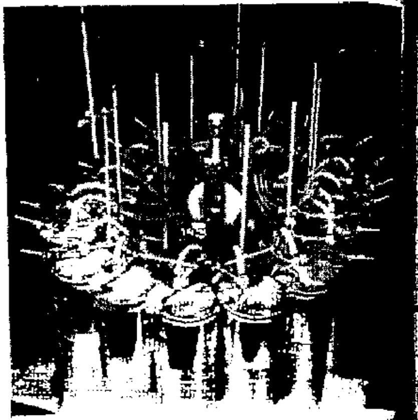

Figure 12. Smoking Machine Set Up to Smo Twenty Cigarettes.

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